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Recovery of Protein-Rich Byproducts from Sweet Potato Stillage following Alcohol Distillation

Y. Victor Wu* and Marvin O. Bagby

Sweet potato can yield 1000 gal of ethanol/acre compared with 250–300 gal/acre for corn. Sweet potatoes of normal, relatively high, and very high dry-matter contents were fermented to ethanol. Pectinase was necessary to decrease viscosity before fermentation for economic processing, especially for varieties of normal and relatively high dry-matter contents. Attained yield of ethanol was 90% of theoretical value. After ethanol was distilled, residual stillage was separated by screening and centrifugation into filter cake, centrifuged solids, and stillage solubles. Filter cake and centrifuged solids had crude protein contents (nitrogen \times 6.25, dry basis) of 22–32% and 42–57%, respectively, and accounted for 44–85% and 0–17% of total sweet potato nitrogen. Sweet potatoes and their fermented products had 4.3–7.6 g of lysine/16 g of N and are expected to have good nutritional value. This practical method to ferment sweet potato for ethanol and to recover valuable protein-rich byproducts may have commercial potential.

Sweet potato is one of the most promising crops for energy production from biomass because it has a long growing season and can continue to increase in weight until it is harvested. The 5.5 tons/acre given in crop production statistics reflects only marketable yields for table use and is much lower than the potential total sweet potato yields. Jones et al. (1983) estimated yields of 570–760 and 712–1140 gal of ethanol/acre for Jewel and HiDry sweet

potatoes, respectively. They believe that potential upper limits are higher than these estimates and that further improvements in dry-matter yields and conversion efficiencies are possible. Azhar and Hamdy (1981) reported alcohol fermentation of sweet potato in a membrane reactor. Matsuoka et al. (1982) carried out alcoholic fermentation of raw sweet potato in a one-step process. Chua et al. (1984) used low-temperature heating or no heating to convert sweet potato starch for ethanol fermentation. Practically no information is available, however, on yield and composition of fermentation residue from sweet potato after ethanol distillation. Optimum use of fermentation residues plays an important role in the commercial success

U.S. Department of Agriculture, Agricultural Research Service, Northern Regional Research Center, Peoria, Illinois 61604.

of all ethanol processes. This paper reports effects of commercial pectinases on viscosities of sweet potato slurries before fermentation and on maximum ethanol concentrations and presents compositions of fermentation products from sweet potatoes with normal (18–24%), relatively high (27–30%), and very high (35% and up) dry-matter contents.

MATERIALS AND METHODS

Sweet Potato. Jewel, Sumor, and HiDry sweet potatoes were from South Carolina. The first two varieties were grown in 1983, and HiDry was grown in 1984. Jewel is the normal commercial sweet potato for table use. Sumor, released in 1984, has relatively high dry-matter and field yields compared to Jewel. HiDry, released in 1984 for industrial use, has very high dry-matter content. Both Sumor and HiDry were tested over several years before official release. Jewel and Sumor were stored under optimal conditions for 8 months before arrival, whereas HiDry was obtained shortly after harvest. Sweet potatoes (around 20 kg for each variety) were ground in a food processor upon arrival and stored at -18°C in food storage bags until use.

Pectinase. CLAREX L and SPARK-L HPG pectinases (Miles Laboratories, Elkhart, IN) are food-grade pectic enzyme systems obtained by the controlled fermentation of *Aspergillus niger*. Pectinol 80SB (Genencor, South San Francisco, CA) is a food-grade pectic enzyme that catalyzes hydrolysis of pectinous materials under mild conditions. KLERZYME LIQUID 200 pectinase (Gist-Brocades, Charlotte, NC) is derived from *A. niger*. Optimum temperatures for CLAREX L, SPARK-L HPG, Pectinol 80SB, and KLERZYME LIQUID 200 are 50, 50, 55, and 60°C , and optimum pH values are 3.5, 3.5, 4.5, and 3.4, respectively.

Fermentation. The viscosity of sweet potato slurry without pectinase treatment was so thick that proper stirring could not be accomplished without significant aqueous dilution. For fermentation without pectinase, minimum amounts of tap water (5750, 6100, 3475 mL) were added to 4500 g of ground wet Jewel, Sumor, and HiDry sweet potatoes, respectively, to form a suitable slurry that can be stirred in a 20-L stainless-steel, temperature-controlled, jacketed fermentor equipped with overhead-drive vane type stirrers. The slurry was adjusted to pH 6.2, and 6 mL of Taka-Therm α -amylase (Miles Laboratories, Elkhart, IN) was added to hydrolyze starch to soluble dextrins. HCl (6 N) and NaOH (12.5 N) were used for pH adjustment. The slurry was maintained at 90°C for 1 h with stirring and then cooled to 60°C by a cooling jacket. Slurry pH was then adjusted to 4.0, and 18 mL of Miles Diazyme L-100 glucoamylase was added to convert dextrins to glucose (2 h with agitation). The mixture was inoculated with 500 mL of yeast (*Saccharomyces cerevisiae*) containing 5 million cells/mL and then fermented at pH 4.5, 30°C . Samples were withdrawn at 0, 24, 48, and 66 h, at which time fermentation was stopped. Additional details of an analogous fermentation procedure were described previously (Wu and Sexson, 1984). Nitrogen from yeast, amylase, and glucoamylase accounted for 7.9–9.8% of total sweet potato nitrogen. Part of the yeast nitrogen is from nucleic acid.

For substrate preparation with pectinase, 0, 1480, and 3670 mL of tap water were added to 11367, 7513, and 5000 g of ground, wet Jewel, Sumor, and HiDry sweet potatoes, respectively, to get a slurry with about 20% dry-matter content. The slurry was adjusted to pH 3.5, and 3 mL of CLAREX L/1000 g of wet sweet potatoes was added. The slurry was stirred 2 h at 50°C to decrease its viscosity.

Subsequently, Taka-Therm α -amylase, Diazyme L-100, and yeast were added as described above. Nitrogen from pectinase, amylase, glucoamylase, and yeast accounted for 4.1–9.4% of total sweet potato nitrogen. This added nitrogen likely has no significant effect on nitrogen determinations of sweet potato fractions.

Fractionation of Stillage. After distillation of alcohol by circulating steam through the outer fermentor jacket, fermentation residue (stillage) was filtered through cheesecloth under suction. Material that retained on the cheesecloth was called filter cake. The thin stillage passing through the cheesecloth was centrifuged at 45200g in a continuous Sharples centrifuge to yield centrifuged solids and stillage solubles. More details were reported previously (Wu and Sexson, 1984). Part of the filter cake was freeze-dried.

Analyses. Protein, fat, fiber, and ash contents were determined by AACC approved methods (1983), and crude protein was calculated from Kjeldahl N \times 6.25. Moisture was determined by heating samples at 100°C to constant weight, and starch was determined by a polarimetric method (Garcia and Wolf, 1972). Nitrogen determinations were made in quadruplicate, moisture in triplicate, and fat and ash in duplicate. Dietary fiber (the sum of cellulose, lignin, and water-insoluble hemicellulose) was determined by the neutral detergent method (McQueen and Nicholson, 1979); in contrast, crude fiber includes only cellulose. Analyses for glucose, fructose, sucrose, glycerol, and ethanol were made with a Waters ALC 200 high-performance liquid chromatograph equipped with refractive index detector (Waters Associates, Milford, MA) on a Bio-Rad HPX-42C (300×7.8 mm, o.d.) column (Richmond, CA) with water eluant.

Amino acid analyses were performed on a Glenco MM-100 amino acid analyzer (Glenco Scientific Inc., Houston, TX) or a Dionex D 300 amino acid analyzer (Dionex Corp., Sunnyvale, CA). After being refluxed in 6 N hydrochloric acid for 24 h, hydrolyzed samples were dried in a rotoevaporator, and residues were dissolved in pH 2.2 citrate buffer. Data were calculated automatically (Cavins and Friedman, 1968). Some sulfur amino acids were determined after oxidation of the sample with performic acid (Moore, 1963).

RESULTS AND DISCUSSION

Effect of Pectinase on Slurry Viscosity. The high viscosity of sweet potato slurries caused stirring problems before fermentation, as well as in processing stillage. Jewel sweet potato slurry has the highest viscosity whereas HiDry has the lowest value at same dry-matter content. CLAREX L and SPARK-L HPG (0.3 and 0.5 mL/100 g of ground, wet sweet potato, respectively) effectively reduced viscosities of sweet potato slurries of up to 25% dry-matter content near pH 3.5 but are not effective at pH 5.6, the unadjusted pH of Jewel sweet potato. Pectinol 80SB and KLERZYME LIQUID 200 (0.1 and 0.3 mL/100 g of ground, wet sweet potato, respectively) were not effective at the unadjusted pH 5.5 of Sumor sweet potato but produced some thinning at optimum pH values of 4.5 (Pectinol 80SB) and 3.4 (KLERZYME LIQUID 200). On the basis of these results, CLAREX L was chosen as the most suitable pectinase for subsequent fermentations, because it is less expensive and more effective at a lower concentration than SPARK-L HPG.

Effect of Fermentation Time on Composition. Table I lists compositions of sweet potato slurries after various fermentation times. Slurries had already been treated with pectinase, α -amylase, and glucoamylase to convert starch and sucrose to glucose. Just before addition of yeast (0

Table I. Effect of Fermentation Time on Sweet Potato Slurry Compositions^a

sweet potato	time, h	compn, g/100 mL				
		sucrose	glucose	fructose	ethanol	glycerol
Jewel	0	2.39	9.76	2.31	0	0.04
	24	0	0	0	6.19	0.54
	48	0	0.01	0	6.33	0.64
	66	0.13	0	0	7.04	0.62
Sumor	0	0.6	16.5	1.4	0	0
	24	0.2	0.1	0	7.8	0.75
	48	0.1	0.1	0	8.0	0.73
	66	0.1	0.1	0	7.9	0.72
HiDry	0	0.38	15.8	1.07	0	0
	24	0.37	0.16	0	6.7	0.63
	48	0.05	0.05	0	7.5	0.78
	66	0.07	0.04	0	7.3	0.74

^a 3 mL of CLAREX L pectinase/1000 g of ground wet sweet potato.

Table II. Effect of Pectinase on Dry-Matter Contents of Wet Sweet Potato Fermentation Residues and Ethanol Concentration

sweet potato (% dry matter)	pectinase ^a	% dry matter of wet		ethanol conc'n, g/100 mL
		filter cake	centrifuged solids	
Jewel (18.3)	no	8.9	31.2	2.5
(20.0)	yes	14.9	30.7	8.0
Sumor (27.4)	no	8.7	32.3	3.75
(26.6)	yes	15.9	26.3	8.0
HiDry (35.6)	no	12.5	38.8	8.0
(35.0)	yes	18.0	31.6	7.5

^a 3 mL of CLAREX L pectinase/1000 g of ground wet sweet potato.

h), glucose was the predominant sugar, although sucrose and fructose were also present. After 24 h of fermentation, almost all sugars disappeared, while ethanol production reached ca. 90% of maximum. Maximum ethanol concentration was reached at 66 h for Jewel and 48 h for Sumor and HiDry. It is possible that some dextrans (not analyzed) converted to glucose and then to ethanol after 48 h for Jewel. Some glycerol was also produced (about one-tenth of the amount of ethanol).

Effect of Pectinase on Dry-Matter Contents of Wet Sweet Potato Fermentation Residues and Ethanol Concentrations. Without pectinase to lower the viscosity of Jewel sweet potato slurry, the maximum ethanol concentration obtained after 66 h of fermentation was 2.5 g/100 mL (Table II). Although almost all starch and sugar were converted to ethanol, the low dry-matter concentration (limited by viscosity) of the slurry caused the low concentration of ethanol. At such a low ethanol concentration, recovery of ethanol by distillation is very expensive. In addition, resulting wet filter cake had only 9% dry matter; it would be costly to dry this fraction. With pectinase the maximum ethanol concentration obtained from Jewel was 8.0 g/100 mL, near that resulting from fermentation of corn and other cereal grains when a similar procedure is used. Dry-matter content of wet Jewel filter cake also increased to 15% when pectinase was added; drying this fraction would be much more economical.

The maximum ethanol concentration resulting from Sumor without pectinase was higher than that of Jewel, but is still low. Addition of pectinase substantially increased resulting ethanol concentration and dry-matter content of Sumor wet filter cake. HiDry, having a very high dry-matter content, can be fermented without pectinase in a slurry containing approximately 20% dry matter and still produce 8 g of ethanol/100 mL. With pectinase, the dry-matter content of wet HiDry filter cake

increased from 12.5 to 18%, which could represent a substantial savings in drying cost.

Yield and Composition of Sweet Potato Fermentation Products. The sweet potato varieties studied contained 5.2–10.1% crude protein, 0.7–0.9% fat, 2.9–5.6% crude fiber, 3.3–4.6% ash, 34.5–68.3% starch, 8.1–22.4% sucrose, 0–6.8% glucose, and 0–5.5% fructose (dry basis) (Table III). Sugar contents of sweet potatoes were determined from freeze-dried samples to prevent possible reaction of sugars with other components at higher drying temperatures. The relatively low starch and high monosaccharide and disaccharide contents of Jewel may be due, in part, to storage: Picha (1986) reported that fresh Jewel contained 1.72% sucrose, 0.09% glucose, and 0.07% fructose and increased in sucrose, glucose, and fructose upon storage. Walter and Hoover (1984) reported fresh Jewel sweet potato had 14.8% starch, 2.4% sucrose, and 0.2% glucose + fructose, whereas after 6 months of storage under optimal conditions Jewel had 8.0% starch, 5.2% sucrose, and 2.8% glucose + fructose. Thus, the sum of starch + sugars decreased from 17.4 to 16.0% for fresh Jewel after 6 months of storage. Walter and Hoover also reported that the percent starch conversion for Jewel decreased with storage. The respective dry matter and starch + sugar (glucose equivalent) for our fresh Jewel before storage were 22.1 and 17.9%, whereas the corresponding values for fresh Sumor were 27.1 and 22.0% (Hamilton and Jones, 1984). The glucose equivalents for Jewel and Sumor were both 81.1% on dry basis before storage (Hamilton and Jones, 1984), whereas our values after storage were 73.9 and 77.3%, respectively. Thus, fresh Sumor has higher dry-matter and glucose equivalents than Jewel before storage, and the difference was greater after storage, because there was no dry-matter loss for Sumor.

Theoretical ethanol yields from 1000 g of fresh sweet potato, calculated from the sum of starch and sugar contents (Table III), were 75, 105, and 151 g for Jewel, Sumor, and HiDry, respectively, whereas the corresponding values for 1983 crops before storage were 92, 112, and 170 (Hamilton and Jones, 1984). Attained ethanol yields for Jewel, Sumor, and HiDry averaged 91, 87, and 93% of theoretical values, respectively. These values were close to those achieved for corn (88%), sorghum (86%), and barley (90%) (Wall et al., 1983; Wu and Sexson, 1984; Wu, 1986).

Fermentation residues, based on initial dry matter, accounted for 36, 32, and 25% of Jewel, Sumor, and HiDry sweet potatoes, respectively; these values were consistent with percent fermentables in Table III. Filter cake accounts for the largest weight of fermentation residue, whereas centrifuged solids were the smallest (Table III). Filter cakes had much higher crude protein, fat, crude

Table III. Yield and Composition of Sweet Potato Fermentation Products^a

product	% of residue	compd, % dry basis								
		protein	fat	crude fiber	ash	starch	sucrose	glucose	fructose	fermentables
Jewel		10.1	0.9	5.6	4.6	34.5	22.4	6.8	5.5	69.2
Jewel FC	45	26.9	3.9	30.9	7.8	0				
Jewel CS	10	48.3	3.0	10.8	3.1	0				
Jewel SS	45	24.1			30.9					
Jewel FC (P)	61	32.4	3.2	19.7	19.3	0				
Jewel CS (P)	0.3	56.6								
Jewel SS (P)	39	14.4			37.9					
Sumor		8.5	0.8	4.3	4.6	58.6	8.1	2.5	1.3	70.5
Sumor FC	61	28.8	2.6	23.3	12.3	0.1				
Sumor CS	7	54.4	2.2	9.5	3.8	0				
Sumor SS	32	17.6			33.1					
Sumor FC (P)	66	29.6	2.7	20.5	12.5	0.9				
Sumor CS (P)	0.2	48.4								
Sumor SS (P)	34	13.8			31.7					
HiDry		5.2	0.7	2.9	3.3	68.3	8.2	0	0	76.5
HiDry FC	75	22.2	3.0	15.9	12.3	3.7				
HiDry CS	2	43.7			4.1					
HiDry SS	24	9.8			27.6					
HiDry FC (P)	53	31.5	3.5	20.3	11.1	3.0				
HiDry CS (P)	1	41.7			5.3					
HiDry SS (P)	46	7.9			35.0					

^a Fermentation residues accounted for 36, 32, and 25% of solids of Jewel, Sumor, and HiDry sweet potatoes, respectively. Key: FC = filter cake; CS = centrifuged solids; SS = stillage solubles; (P) = 3 mL pectinase added/1000 g of wet sweet potato before fermentation. Sumor, Sumor FC (P), HiDry, and HiDry FC (P) had 6.1, 29.4, 3.7, and 40.1% neutral detergent fiber, respectively.

Table IV. Amino Acid Composition of Sweet Potatoes and Their Fermented Products (g of Amino Acid/16 g of N)^a

	sweet potato			filter cake			centrifuged solids			stillage solubles		
	J	S	H	J	S	H	J	S	H	J	S	H
aspartic acid	22.0	19.5	18.8	17.2	15.9	14.1	16.1	16.0	15.0	25.7	15.5	13.0
threonine	5.1	5.9	5.5	6.5	5.6	5.7	5.9	6.2	6.5	5.8	7.4	6.8
serine	5.0	5.2	6.2	6.5	5.7	6.2	6.3	6.3	6.7	5.4	6.3	6.0
glutamic acid	8.8	10.4	8.0	10.4	10.6	11.1	10.5	12.8	11.6	15.3	11.6	14.8
proline	2.8	4.5	2.9	4.3	7.5	5.1	4.4	3.2	4.4	5.8	3.8	9.9
glycine	3.6	4.0	4.4	5.4	5.4	5.3	5.1	5.4	5.8	5.3	8.5	10.8
alanine	4.5	4.7	5.6	5.8	5.9	6.0	6.1	6.7	6.5	6.9	7.6	7.1
valine	5.2	6.0	6.0	7.4	6.6	7.2	7.5	6.9	7.5	4.4	5.9	4.9
half-cystine	0.6	0	0.7 (2.0)	1.4	0	0.6 (1.3)	1.2	0	0.5	0	0	1.7 (2.4)
methionine	0.9	1.3	0.8 (1.5)	0.7	1.8	0.4 (1.5)	2.3	1.8	0	0.7	1.3	0.3 (0.3)
isoleucine	3.5	4.2	4.3	5.2	5.1	5.1	5.4	5.5	6.1	2.9	4.8	3.7
leucine	5.4	6.1	6.4	8.1	7.7	8.7	8.1	8.8	9.0	3.2	5.7	4.4
tyrosine	3.5	4.0	3.9	5.3	5.1	4.8	5.2	5.3	5.1	2.0	2.2	2.4
phenylalanine	4.8	5.8	6.1	6.5	6.3	6.0	6.1	6.5	6.2	2.5	3.9	3.2
lysine	4.3	4.8	4.5	6.5	6.6	5.9	7.0	7.6	7.6	4.8	6.8	6.4
histidine	2.1	2.2	2.1	2.6	2.2	2.6	2.5	2.2	2.5	1.6	2.1	2.2
arginine	5.9	6.7	4.9	6.0	6.0	6.2	6.2	6.3	6.1	2.0	3.1	4.1

^a Tryptophan not determined. Values in parentheses are determined after performic acid oxidation. Key: J = Jewel; S = Sumor; H = HiDry.

fiber, neutral detergent fiber, and ash contents than the original sweet potatoes. Crude protein contents of filter cake increased when pectinase was added; HiDry filter cake showed the largest increase (22–32%). Since the filter cake contained less stillage solubles (lower crude protein content than filter cake) when pectinase was added (Tables II and III), higher crude protein content for filter cake resulted. Stillage solubles had highest ash contents; part of this ash results from pH adjustments before fermentation. Pectinase decreased the percentage of centrifuged solids drastically, especially for Jewel and Sumor.

Amino Acid Composition. Table IV presents amino acid compositions of sweet potatoes and their fermented products. In general, the three sweet potatoes have similar amino acid compositions. Jewel sweet potato had 4.3 g of lysine/16 g of N compared with 6.6 and 3.8 reported by Purcell and Walter (1982) and Walter et al. (1983), respectively, for the same variety. Filter cake, centrifuged solids, and stillage solubles all had higher lysine contents than the sweet potato varieties themselves. Aspartic acid

and glutamic acid (or their amide forms) have the highest percentage among all amino acids in all sweet potatoes and fermented fractions. Because some half-cystine and methionine may be destroyed by acid hydrolysis, half-cystine and methionine were also determined after performic acid oxidation for HiDry, HiDry filter cake, and HiDry stillage solubles; in general, higher values were obtained.

There was also little difference between amino acid compositions of freeze-dried and 80 °C dried Jewel and Sumor sweet potatoes (data not shown). Apparently, the drying temperatures used here did not affect amino acid compositions.

CONCLUSIONS

Corn is the major biomass currently used for ethanol production (Morris, 1983). Optimal use of the fermentation residue, after ethanol is distilled, is important for success of commercial ethanol processes. Other biomass types such as sweet potatoes may be feasible alternative raw materials for ethanol production, however. For such

utilization, it is necessary to optimize the fermentation process itself (which represents, for sweet potato, a problem due to viscosity of resulting slurries) and to estimate the value of resulting protein-rich byproducts.

Addition of pectinases to sweet potatoes benefits economic recovery of ethanol from varieties of normal and relatively high dry-matter contents. Pectinase also increases the solids content of wet filter cake for all sweet potato varieties and decreases drying costs of wet filter cake since less water needs to be removed for a fixed amount of dry matter. In addition, pectinase increases crude protein contents of sweet potato filter cake and decreases amounts of centrifuged solids to such a degree that centrifugation can be eliminated. Of the varieties examined, HiDry sweet potato, having very high dry-matter content, appears most desirable for ethanol fermentation, since less fresh sweet potato is needed for a fixed amount of ethanol production.

Sweet potato filter cake may have food potential, since it has high crude protein content and an amino acid balance superior to that of cereal grains. The relatively low fat content of sweet potato filter cake, compared with that of corn distillers' grains, may improve storage stability.

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